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Preliminary X-ray investigation of pseudo-wollastonite.

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Pseudo-wollastonite ($CaSiO_3$), sometimes also described as α and, more rarely, as β wollastonite, is the stable form of wollastonite above about 1126° C. It was originally discovered by Breñosa (1885) in a devitrified glass of doubtful origin and named 'Bourgeoisita'. It occurs commonly in melts, slags and some glasses and has been found naturally in metamorphosed rocks in South West Persia (McLintock, 1932). Pseudo-wollastonite usually crystallizes as small, irregular grains, often tabular, but sometimes also as fibres arranged in parallel or divergent groups. Polysynthetic twinning is common. The crystals give rise to a nearly uniaxial interference figure, are optically positive and strongly birefringent; $\alpha = 1{\cdot}610$, $\beta = 1{\cdot}611$, $\gamma = 1{\cdot}654$; $2V = 9°$ (Ferguson & Merwin, 1919). X-ray powder data have been recorded (e.g. Clark, 1946; A.S.T.M. index) but no single-crystal X-ray work has been described. The powder data are in agreement with those obtained in the present investigation, which, together with the powder data of other anhydrous and hydrated calcium silicates, are being compiled for publication elsewhere.

The crystals for the present investigation were kindly supplied by the Building Research Station. They were prepared by heating a stoichiometric mixture of pure calcium carbonate and pure ground quartz at 1550° C. and cooling slowly to about 1000° C. The crystals occurred as shiny flat platelets on the surface of the melt, the largest single crystals being about $250 \times 250 \times 10\ \mu$. Their optical properties were in agreement with those quoted above, with γ perpendicular to the plate.

X-ray oscillation, rotation and Weissenberg photographs were taken about an axis approximately perpendicular to the plate (c) and about two axes in the plate at right angles to each other. The cell dimensions thus deduced were

$$a = 6{\cdot}90,\ b = 11{\cdot}78,\ c = 19{\cdot}65\ \text{Å}\,,$$
$$\alpha = \gamma = 90°,\ \beta = 90°\ 48'\,.$$

The density determined at the Building Research Station was $2{\cdot}92$ g.cm.$^{-3}$, which gives 24 formula units of $CaSiO_3$ per unit cell (calculated density $2{\cdot}90$ g.cm.$^{-3}$).

The cell is triclinic but has pseudo-planes of symmetry perpendicular to all three crystallographic axes and is thus pseudo-orthorhombic. Although the single crystals available were too small to be easily tested for piezoelectricity, several polycrystalline pieces were found containing larger single individuals which were suitable for this purpose. No piezoelectricity could be detected. Even the largest fragments proved too small to be tested for pyro-electricity. The absence of a centre of symmetry was therefore not established.

Oscillation photographs of a crystal mounted about c, taken at 60° and 120° to each other, as well as Laue photographs with the X-ray beam parallel to c, showed c to be a pseudohexagonal axis. The $00l$ reflexions were absent for l odd; c is therefore a pseudo-6_3 axis. The pseudo-hexagonality accounts for the nearly uniaxial optic figure.

Systematic absences were observed for all hkl reflexions for which $h+k$ was odd, i.e. the cell is C face-centered. A primitive cell could therefore be chosen half the size of the original cell. The smaller cell has dimensions

$$a' = b' = 6{\cdot}82,\ c' = 19{\cdot}65\ \text{Å}\ (= c)\,,$$
$$\alpha' = \beta' = 90°\ 24',\ \gamma' = 119°\ 18'\,.$$

It retains the pseudo-hexagonal c axis and departs from a hexagonal outline by only a few minutes in each of the angles, but is triclinic.

Some additional regularities observed in the diffraction pattern are more conveniently described in terms of the larger pseudo-orthorhombic cell, to which all subsequent data are therefore referred.

In addition to the systematic absences for all hkl reflexions when $h+k = 2n+1$, absences were also observed for all $0kl$ reflexions for which $l = 4n+2$. Each half cell $a;\ b;\ \frac{1}{2}c$ thus has a pseudo-c glide plane perpendicular to a, but the whole cell has no such overall glide plane.

Layer lines corresponding to $k = 3n$ on rotation photographs about the b axis were much stronger than the rest. (As expected from the pseudo-hexagonality, a rotation photograph about an axis at 60° to b in the $a\,b$ plane closely resembled the rotation photograph about b.) If the weaker layer lines are neglected, there is a true plane of symmetry perpendicular to b, so that each sub-cell ($a'' = a;\ b'' = \frac{1}{3}b;\ c'' = c$) is monoclinic. Moreover, each sub-cell, like the original pseudo-orthorhombic one, is c face-centered and shows a pseudo-c glide plane perpendicular to a'' in each half-cell $a'';\ b'';\ \frac{1}{2}c''$. The sub-cells are more strongly pseudo-hexagonal than the true cell, with hexagonal axes $a''' = b''' = \frac{1}{3}b;\ c''' = c$. The relationship between the four cells is shown in Fig. 1.

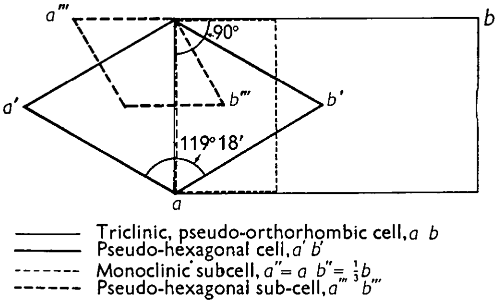

Fig. 1. The relationship between the unit cells. c is the same for all four cells.

It was impracticable to proceed with a detailed structure determination, but it was thought possible that some structural resemblance might be found between the high- and low-temperature forms of wollastonite which would facilitate further investigations. Single crystals of the two forms were therefore heated at temperatures below and above the transition temperature, respectively. The transformation of either modification into the other was sluggish, requiring several days' heating, but the crystals did not shatter. X-ray photographs of the products showed that both transformations gave rise to a random array of small crystallites which were either produced directly by breaking-up of the larger original crystal or by a complete disintegration of the original structure, followed by recrystallization. In either case the experiments do not demonstrate any structural resemblance between the two modifications which could be used to elucidate one of the crystal structures with the help of the other.

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The crystal structure of succinamide, $(CH_2CONH_2)_2$.*

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A considerable amount of accurate information has been collected about the configuration and dimensions of the peptide amide group, whereas comparable data for the terminal amide group are still missing. Only three crystal structure determinations of compounds containing the terminal amide group have been published, namely, those of acetamide (Senti & Harker, 1940), L-glutamine (Cochran & Penfold, 1952) and glycyl-L-asparagine (Pasternak, Katz & Corey, 1954; see also Katz, Pasternak & Corey, 1952); the dimensions reported for the group are appreciably different. For this reason an accurate crystal structure analysis of a simple amide would be of interest. The crystals of succinamide appeared to be specially suitable for a study, because of the simple molecular structure of this compound and also because a planar configuration was expected for its molecule by analogy to related compounds. In crystals of succinic acid the molecule is extended and planar (MacGillavry, Hoogschagen & Sixma, 1948; Morrison & Robertson, 1949), and the same holds for the succinamic acid part of glycyl-L-asparagine. If the molecule of succinamide also is extended and planar, then each of the two C–N bonds and the central C–C bond have to be *trans* for steric reasons, and the molecule has, like the molecule of succinic acid, a center of symmetry.

Crystals of succinamide (decomposition point about 250° C.) were easily obtained from water in monoclinic plates bounded by (001) faces. Perfect cleavage along (100) indicated a layer structure parallel to this plane. The unit-cell dimensions derived from rotation and Weissenberg photographs are

$$a_0 = 6{\cdot}96,\ b_0 = 8{\cdot}02,\ c_0 = 9{\cdot}90\ \text{Å},\ \beta = 102{\cdot}5°;$$

density (by flotation) = $1{\cdot}442$ g.cm.$^{-3}$; number of molecules $n = 4$ (calc. 3·98); systematic absences, hkl when $h+k$ is odd, $h0l$ when h and l are odd. These absences are characteristic for the space groups Cc ($n = 4$) and $C2/c$ ($n = 8$). Since there are only four molecules in the unit cell, this latter centrosymmetric space group would require the molecule to lie on a center of symmetry.

In addition to the systematic absences, very pronounced pseudo-absences are observed in the ($hk0$) zone; reflections with h and k both odd are missing except two which are extremely weak. By contrast, the ($h00$) reflections are outstandingly strong and show the normal decline. This intensity pattern is compatible only with a layer structure parallel to (100) with all the atoms lying almost exactly in the planes $x = \frac{1}{4}$ and $x = \frac{3}{4}$; the

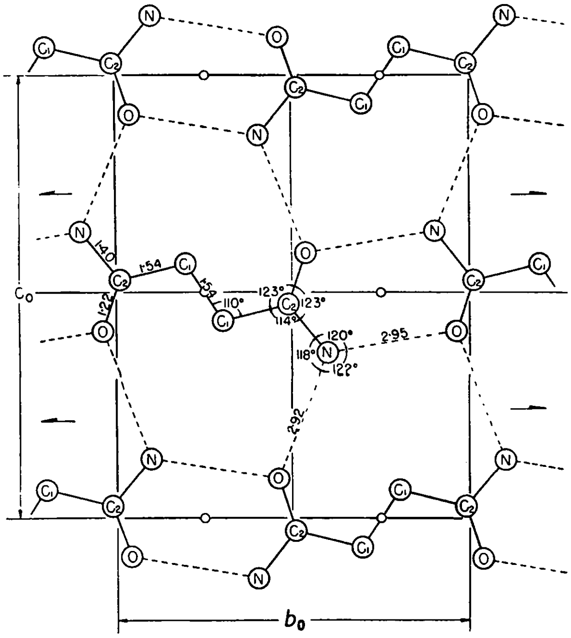

Fig. 1. The arrangement of the molecules of succinamide within the layer $x = \frac{1}{4}$ or $x = \frac{3}{4}$.

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